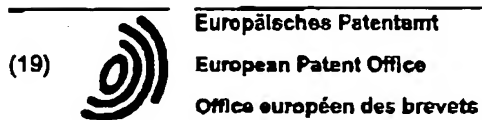


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(54) **Flow divider for analytical purposes**
Fluss-Verteiler zu analytischen Zwecken
Diviseur d'écoulement à usage analytique

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JP-A- 59 100 312 **US-A- 4 800 754**
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EP 1 248 096 B1

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1

EP 1 248 096 B1

2

Description

[0001] The present invention relates to a flow divider for dividing a fluid flow into a number of fluid flows, in particular for analytical or preparative fluid measurement technology and/or for micro-fluid systems, which has at least one working sensor assigned to one of the fluid flows and which comprises a control unit for regulating the pressure of one of the fluid flows and/or one of the flow rates of the fluid flows, which is coupled to one or each working sensor and an actuator for altering the flow rate of this fluid flow.

[0002] Flow dividers designated as splitters are used in analytical or preparative fluid measurement technology, in particular in association with devices for generating and supplying fluid volume flows in capillaries, preferably in chromatographic separating columns for analytical fluid separating technology. A first application comprises dividing the entire flow generated and supplied by a pump into at least two partial flows, an excess flow in an excess path and a working flow in a working path. In the process the desired working flow in the separating column is adjusted and provided by means of so-called restrictors, that is, by hydraulic resistors arranged in the excess path. To maintain the working flow, that is, the volume flow flowing through the capillaries, substantially constant, depending on the pressure conditions and/or volume conditions in the working path changing as a result of disturbances, for example, DE 199 14 358 A1 discloses a device and a process for providing fluid volume flows in capillaries, which exhibits at least one working sensor and a control unit for regulating the working flow and/or the pressure in the working path, whereby the control unit is coupled to the working sensor and a means for altering the working flow. This device enables the pressure and/or the working flow to be measured and kept constant advantageously in the working path, yet allows this device no corresponding possibilities and measures in the other splitter branch, that is, the excess path in this case.

[0003] Another preferred application of such splitters in analytical or preparative fluid measurement technology and/or micro-fluid technology comprises splitting the fluid coming through the separating column or the separating channel into two or more fluid flows, so that these can be supplied to fraction collectors and/or mass spectrometer detectors arranged downstream. Passive splitters are used chiefly for this, that is, individual elements which exhibit a different hydraulic flow resistance. Splitters with back pressure controllers have also become known, whereby the back pressure is kept constant in one of the split branches only. Individually it cannot be avoided that the volume flow changes depending on the physical properties of the fluid and the fluctuations in pressure in each other splitter.

[0004] It is accordingly an aim of the invention to make available a flow divider having a reduced back pressure sensitivity, enabling the splitting ratio to be kept constant

independently of the physical properties of the fluid and/or the fluctuations in pressure.

[0005] This task is solved according to the present invention by the features of Claim 1.

[0006] With the flow divider according to the present invention the volume of the splitter branches can be adjusted according to the splitting ratio such that the fluid front respectively progresses parallel. In this way media-specified breakdowns occur isochronously so that they can be compensated.

[0007] The foregoing measures enable the split ratio between each two of the fluid flows to be precisely specified and/or be kept constant. By at least a number of the working sensors assigned to the affected fluid flows corresponding to the number of divided fluid flows being provided, the splitter according to the present invention can be operated independent of media, that is, the split ratio and/or the volume flow can be kept constant in at least one splitter branch independently of the physical properties of the fluid. In contrast to the passive splitters known from the prior art, in which the split ratio is either unknown or detectable only by expensive calibrating, the splitter according to the present invention concerns an active splitter. Via the abovementioned measures the principal drawback of the passive splitters known from the prior art, namely the back pressure sensitivity, can be decreased or completely eliminated. Such active splitters are also distinguished by a greater application flexibility, as they can be employed in all areas where a number of partial flows is to be split off from a single fluid flow.

[0008] With the flow divider according to the present invention and realised as an active splitter a fluid flow or influx can be divided into two to n partial flows. The ratio of the individual partial flows can either be firmly preset or adjusted according to the user and application requirements. Typical values for the splitter ratios realisable with such active splitters are 1:1 to 1:10000. It is understood that n-1 partial flows can also be split off using the flow divider according to the present invention.

[0009] The number, arrangement and type according to the present invention of the working sensors enable different properties of the fluids flowing through the flow divider, for example their viscosity or thermal capacity, to be compensated advantageously and calculated by means of a computer unit coupled to the control unit. This is particularly beneficial because for the preferred flow region between approximately 10 nl/min to approximately 1000 ml/min, in particular from 1 nl/min to 100 ml/min, no direct measuring processes independent of the properties of the fluids are known to date and because frequently calibration is not possible, as the fluid mixtures being worked with are unknown.

[0010] If the working sensors are designed as flow sensors directly measuring the fluid flow, in particular as thermal mass flow rate meters, a comparatively simple structure for the flow divider and a comparatively simple computer evaluation of the measurement signals is en-

3

EP 1 248 096 B1

4

abled, because in this case the splitter ratio can be determined directly by simple relational development of the measuring values.

[0011] In the case where the flow divider is provided with such working sensors whose measurement signals depend on the physical properties of the fluid flowing through the flow divider and whose flow rate or volume flow is to be determined, working sensors are to be provided which are of identical design in this respect.

[0012] If at least one of the working sensors is assigned advantageously to each of the divided fluid flows, the splitting ratio can be determined directly and/or kept constant particularly easily by this.

[0013] If an adjustable actuator for changing the respective fluid flow is assigned to each of the divided fluid flows, the splitting ratio and thus the volume flow can be kept constant in each branch. Sample analysis can be performed parallel in each branch by this, enabling a particularly economical method of operation.

[0014] It is effective, if the actuator or each actuator exhibits a continuously changeable hydraulic flow resistance. Electromagnetic regulating valves and/or temperature-controlled actuators can be used, by way of example. Independently of whether variable restrictors adjustable step by step or continuously adjustable are used, they must have an adequate dynamic range, that is, the flow resistance must be adjustable in correspondingly broad limits in order to be able to equalise the possible external fluctuations in back pressure, dependent of the respective applications, by corresponding change of the hydraulic resistance.

[0015] According to a preferred field of application the flow divider is effectively arranged such that it enables division into fluid flows in a range of application or working sphere of approximately 10 ml/min to approximately 1000 ml/min, whereby applications to a microrange of approximately 1 nl/min are conceivable, if required.

[0016] The present task is solved also by a process for dividing a fluid flow into a number of divided fluid flows as claimed in Claim 5.

[0017] The abovementioned characteristics contribute both individually and in any combination to diminishing the back pressure sensitivity when one fluid flow is divided into a number of fluid flows, and enable the volume flow to be kept constant independent of the physical properties of the fluid assigned thereto and the fluctuations in pressure in this fluid in at least one splitter branch.

[0018] Further features, viewpoints and advantages of the invention will emerge from the following description, wherein two preferred embodiments of the invention are described in greater detail with reference to the figures, in which:

Figure 1 is a diagrammatic view of a first embodiment of the invention having a flow divider arranged as a 1- to 2-way splitter;

Figure 2 is a diagrammatic view of a second embodi-

ment of the invention having a flow divider arranged as a 1- to 4-way splitter.

[0019] Figure 1 illustrates flow divider 20 according to a first embodiment of the invention, which is arranged here as a 1- to 2-way splitter. This means that flow divider 20 divides inflowing fluid flow 30 into 2 partial flows, designated here as fluid flow 31 and fluid flow 32. The divided fluid flows 31 and 32 are accordingly assigned to a first splitter branch 33 and a second splitter branch 34. Disposed in first splitter branch 33 is a working sensor 41 designed as a flow sensor and an actuator 51 is arranged downstream, whereas only one working sensor is arranged as flow sensor 42 in second splitter branch 34. Working sensors 41 and 42 as well as actuator 51 are coupled by means of electrical wires for example to a control unit 55, as illustrated in Figure 1 by the dashed lines. In the embodiment illustrated control unit 55 comprises a computer unit. The computer unit serves to calculate the splitter ratio, that is, the ratio of the volume rates of fluid flow 31 and of fluid flow 30 from the measurement signals detected by working sensors 41 and 42. The result is converted in control unit 55 into a corrective signal for an actuator 51. Depending on the self-adjusting back pressure downstream of actuator 51 in first splitter branch 33 fluid flow 31 is changed and consequently fluid flow 32 also, whereby the respective changes are detected by working sensors 41 and 42 and forwarded to control unit 55. This control unit 55 regulates fluid flows 31 and 32 using an appropriate algorithm such that the ratio between both these fluid flows remains substantially constant. In the embodiment of the 1- to 2-way splitters this means that by necessity fluid flow 31 is regulated to an essentially constant value, as inflowing fluid flow 30 can be assumed to be constant. An example of a typical splitting ratio between fluid flows 31 and 32 is 1/1000.

[0020] In the embodiment of a flow divider 60 illustrated in Figure 2 this is designed as a 1- to 4-way splitter. Inflowing fluid flow 70 is divided up into four partial flows, designated here as fluid flows 71, 72, 73, 74. The splitting ratio is typically adjusted such that a fluid flow 71, 72, 73, 74, which accounts for a quarter of the value of fluid flow 70, is realised in each of the four assigned splitter branches 75, 76, 77, 78. Otherwise expressed, the splitter ratio is a quarter in each of individual splitter branches 75, 76, 77, 78.

[0021] Each of the four splitter branches 75, 76, 77, 78 comprises a working sensor 81, 82, 83, 84 designed as flow sensor to which an actuator 91, 92, 93, 94 is respectively connected downstream. Likewise for flow divider 20 of the first embodiment according to Figure 1, with the second embodiment of flow divider 60 according to Figure 2 working sensors 81, 82, 83, 84 are realised identically with respect to the dependence of their measurement signals on the physical properties of the fluid to be measured. This means that when a change is made to the physical properties of the fluid in

5

EP 1 248 096 B1

6

respective splitter branches 33, 34; 75, 76, 77, 78 the respective relative changes of the measurement signals of individual flow sensors 41, 42; 81, 82, 83, 84 proceed identically in strength and direction. This enables the volume flow to be determined and/or kept constant in individual splitter branches 33, 34; 75, 76, 77, 78 or the respective splitting ratios to be determined and/or kept constant independently of the respective physical properties of the fluid and/or the fluctuations in pressure in respective splitter branches 33, 34; 75, 76, 77, 78 by ratio development or calculation in the computer unit of control unit 55.

[0022] Both flow divider 20 and flow divider 60 are realised as active splitters. This means that said splitters 20, 60 are able to eliminate the main disadvantage of passive splitters common in the prior art, namely a sensitivity to back pressure in the respective splitter branches. The flow dividers are preferably designed as a separately manageable unit.

[0023] Likewise for actuator 51 of flow divider 20 according to the first embodiment with actuators 91, 92, 93, 94 of flow divider 60 according to the second embodiment, it is a case of variable restrictors, that is, adjustable actuators which are variable with respect to their hydraulic flow resistance. The hydraulic resistance of the respective actuators can be changed preferably continuously, thus enabling constant regulation of the volume flow or flows.

[0024] Both working sensors 81, 82, 83, 84 and actuators 91, 92, 93, 94 are coupled to control unit 95 by electrical wires, by way of example, as illustrated in Figure 2 by way of dashed lines. It is understood, however, that the coupling may be in any form, in particular in the form of electromagnetic waves or similar.

[0025] In accordance with a preferred application of flow dividers 20, 60 they are used in analytical or preparative fluid separation technology either for dividing a whole flow conveyed by a supply device, in particular a pump, into an excess flow in an excess path and a working flow in a working path, wherein a separating mechanism, in particular a separating column, is arranged, or flow dividers 20, 60 are arranged downstream in the working path after the separating mechanism, in particular after the separating column. The working flow is divided up by flow dividers 20, 60 into at least two or more fluid flows 31, 32 or 71, 72, 73, 74, therefore into at least two or more splitter branches 33, 34 or 75, 76, 77, 78. After they pass working sensors 41, 42; 81, 82, 83, 84 and actuators 51; 91, 92, 93, 94 fluid flows 31, 32; 71, 72, 73, 74 divided into corresponding splitter branches 33, 34; 75, 76, 77, 78 are directed to appropriate detectors which can also be designed as mass-spectrometer detectors. Fraction collectors, in which the fluids or fluid mixtures to be collected can be collected according to content, can be attached to the detectors in an application in the area of preparative fluid separation technology.

LIST OF REFERENCE NUMBERS

[0026]

5	20	Flow divider
	30	Fluid flow
	31	Fluid flow
	32	Fluid flow
	33	First splitter branch
10	34	Second splitter branch
	41	Working sensor
	42	Working sensor
	51	Actuator
	55	Control unit
15	60	Flow divider
	70	Fluid flow
	71	Fluid flow
	72	Fluid flow
	73	Fluid flow
20	74	Fluid flow
	75	First splitter branch
	76	Second splitter branch
	77	Third splitter branch
	78	Fourth splitter branch
25	81	Working sensor
	82	Working sensor
	83	Working sensor
	84	Working sensor
	91	Actuator
30	92	Actuator
	93	Actuator
	94	Actuator
	95	Control unit

Claims

1. A flow divider for dividing a fluid flow into a number of divided fluid flows, in particular for analytical or preparative fluid measurement technology and/or for micro-fluid systems, which has at least one working sensor assigned to one of the divided fluid flows and which comprises a control unit for regulating the pressure of one of the fluid flows and/or one of the flow rates of the fluid flows, which is coupled to each working sensor and an actuator for altering the flow rate of this fluid flow, and
 at least two working sensors (41, 42; 81, 82, 83, 84) are provided, wherein at least one of the working sensors (41, 42; 81, 82, 83, 84) is assigned to each of the divided fluid flows (31, 32; 71, 72, 73, 74), characterized in that the working sensors (41, 42; 81, 82, 83, 84) are designed as thermal mass flow rate meters which are designed identically with respect to the dependence of their measurement signals on the physical properties of the fluid flowing through the flow divider, so that a compensation of

7

EP 1 248 096 B1

8

the effect of the dependence of the measurement signals of the working sensors (41, 42; 81, 82, 83, 84) on the physical properties of the fluid is enabled by way of a ratio development of the measurement signals of the working sensors (41, 42; 81, 82, 83, 84).

2. A flow divider as claimed in Claim 1, characterised in that at least one of the working sensors (81, 82, 83, 84) and at least one actuator (91, 92, 93, 94) for altering the flow rate of the respective fluid flow rate are assigned to each of the divided fluid flows (71, 72, 73, 74) and are coupled to the control unit (95).
3. Flow divider as claimed in one of Claims 1 or 2, characterised in that the actuator (51; 91, 92, 93, 94) exhibits a continuously changeable hydraulic flow resistance.
4. Flow divider as claimed in any one of Claims 1 to 3, characterised in that it enables separation into fluid flows (31, 32; 71, 72, 73, 74) in a range of 10 nl/min to 1000 ml/min.
5. A process for dividing a fluid flow into a number of divided fluid flows, in particular in analytical or preparative fluid measurement technology and/or in micro-fluid systems, wherein a control unit which is coupled to at least one working sensor, which is assigned to one of the divided fluid flows, and to an actuator for changing this fluid flow, regulates the pressure of one of the fluid flows and/or regulates one of the flow rates of the fluid flows, and wherein by means of at least two working sensors (41, 42; 81, 82, 83, 84) the respective flow rate of the fluid flows (31, 32; 71, 72, 73, 74) is measured characterized in that said working sensors are designed as thermal mass flow rate meters which are designed identically with respect to the dependence of their measurement signals on the physical properties of the fluid flowing through the flow divider, of which respectively at least one is assigned to each of the divided fluid flows (31, 32; 71, 72, 73, 74), wherein the effect of the dependence of the measurement signals of the working sensors (41, 42; 81, 82, 83, 84) on the physical properties of the fluid is compensated by way of a ratio development of the measurement signals of the working sensors (41, 42; 81, 82, 83, 84), and wherein the control unit regulates the flow rates of the fluid flows (31, 32; 71, 72, 73, 74) such that the ratio between at least two of the flow rates of the fluid flows (31, 32; 71, 72, 73, 74) remains constant.

Patentansprüche

1. Stromteiler zum Aufteilen eines Flüssigkeitsstroms in eine Anzahl aufgeteilter Flüssigkeitsströme, insbesondere für die analytische oder präparative Flüssigkeitsmesstechnik und/oder für Mikrofluidsysteme, wobei der Stromteiler mindestens einen Arbeitssensor aufweist, der einem der aufgeteilten Flüssigkeitsströme zugeordnet ist, und eine Regelungsvorrichtung zum Regeln des Drucks eines der Flüssigkeitsströme und/oder der Flussraten der Flüssigkeitsströme umfasst, welche mit jedem Arbeitssensor und einem Stellglied zur Änderung der Flussrate dieses Flüssigkeitsstroms verbunden ist, und wobei mindestens zwei Arbeitssensoren (41, 42; 81, 82, 83, 84) bereitgestellt werden, wobei mindestens einer der Arbeitssensoren (41, 42; 81, 82, 83, 84) jedem der aufgeteilten Flüssigkeitsströme (31, 32; 71, 72, 73, 74) zugeordnet ist, dadurch gekennzeichnet, dass die Arbeitssensoren (41, 42; 81, 82, 83, 84) bezüglich der Abhängigkeit ihrer Messsignale von den physikalischen Eigenschaften der durch den Stromteiler strömenden Flüssigkeit als thermische Massenflussratenmesser gleicher Bauart ausgeführt sind, sodass die Wirkung der Abhängigkeit der Messsignale der Arbeitssensoren (41, 42; 81, 82, 83, 84) von den physikalischen Eigenschaften der Flüssigkeit durch Einstellen eines Verhältnisses der Messsignale der Arbeitssensoren (41, 42; 81, 82, 83, 84) kompensiert werden kann.
2. Stromteiler nach Anspruch 1, dadurch gekennzeichnet, dass mindestens einer der Arbeitssensoren (81, 82, 83, 84) und mindestens ein Stellglied (91, 92, 93, 94) zur Änderung der Flussrate der jeweiligen Flüssigkeit jedem der aufgeteilten Flüssigkeitsströme (71, 72, 73, 74) zugeordnet und mit der Regelungsvorrichtung (95) verbunden sind.
3. Stromteiler nach einem der Ansprüche 1 oder 2, dadurch gekennzeichnet, dass das Stellglied (51; 91, 92, 93, 94) einen kontinuierlich veränderbaren Flüssigkeitsströmungswiderstand aufweist.
4. Stromteiler nach einem der Ansprüche 1 bis 3, dadurch gekennzeichnet, dass dieser die Aufteilung in Flüssigkeitsströme (31, 32; 71, 72, 73, 74) in einem Bereich von 10 nl/min bis 1000 ml/min ermöglicht.
5. Verfahren zum Aufteilen eines Flüssigkeitsstroms in eine Anzahl aufgeteilter Flüssigkeitsströme, insbesondere in der analytischen oder präparativen Flüssigkeitsmesstechnik und/oder in Mikrofluidsystemen, wobei eine mit mindestens einem Arbeitssensor, der einem der aufgeteilten Flüssigkeitsströme zugeordnet ist, und einem Stellglied zur Änderung dieses Flüssigkeitsstroms verbundene Regel-

9

EP 1 248 096 B1

10

vorrichtung den Druck eines der Flüssigkeitsströme und/oder eine der Flussraten der Flüssigkeitsströme regelt, und wobei mittels mindestens zweier Arbeitssensoren (41, 42; 81, 82, 83, 84) die entsprechenden Flussraten der Flüssigkeitsströme (31, 32; 71, 72, 73, 74) gemessen werden, dadurch gekennzeichnet, dass die Arbeitssensoren (41, 42; 81, 82, 83, 84) bezüglich der Abhängigkeit ihrer Messsignale von den physikalischen Eigenschaften der durch den Stromteiler strömenden Flüssigkeit als thermische Massenflussratenmesser gleicher Bauart ausgeführt sind, wobei jedem der aufgeteilten Flüssigkeitsströme (31, 32; 71, 72, 73, 74) mindestens ein Arbeitssensor zugeordnet ist, wobei die Wirkung der Abhängigkeit der Messsignale der Arbeitssensoren von den physikalischen Eigenschaften der Flüssigkeit durch Einstellen eines Verhältnisses der Messsignale der Arbeitssensoren (41, 42; 81, 82, 83, 84) kompensiert werden kann und die Regelvorrichtung die Flussraten der Flüssigkeitsströme (31, 32; 71, 72, 73, 74) so regelt, dass das Verhältnis zwischen mindestens zwei der Flussraten der Flüssigkeitsströme (31, 32; 71, 72, 73, 74) konstant bleibt.

Revendications

1. Diviseur d'écoulement pour diviser un écoulement de fluide en un certain nombre d'écoulements de fluide divisés, en particulier pour la technologie des mesures d'un fluide analytique ou préparatif et/ou pour des systèmes microfluides, qui possède au moins un détecteur de travail attribué à un des écoulements de fluide divisés et qui comprend une unité de commande pour réguler la pression d'un des écoulements de fluide et/ou un des débits des écoulements de fluide, qui est couplé à chaque détecteur de travail et à un actionneur pour modifier le débit de cet écoulement de fluide, et au moins deux détecteurs de travail (41, 42; 81, 82, 83, 84) sont fournis, dans lesquels au moins un des détecteurs de travail (41, 42; 81, 82, 83, 84) est attribué à chacun des écoulements de fluide divisés (31, 32; 71, 72, 73, 74), caractérisé en ce que les détecteurs de travail (41, 42; 81, 82, 83, 84) se présentent sous forme de débitmètres massiques thermiques qui sont conçus de façon identique en ce qui concerne la dépendance de leurs signaux de mesure vis-à-vis des propriétés physiques du fluide coulant à travers le diviseur d'écoulement, de sorte qu'une compensation de l'effet de la dépendance des signaux de mesure des détecteurs de travail (41, 42; 81, 82, 83, 84) vis-à-vis des propriétés physiques du fluide est obtenue au moyen d'un développement proportionnel des signaux de mesure des détecteurs de travail (41, 42; 81, 82, 83, 84).

2. Diviseur d'écoulement selon la revendication 1, caractérisé en ce qu'au moins un des détecteurs de travail (81, 82, 83, 84) et au moins un actionneur (91, 92, 93, 94) pour modifier le débit d'écoulement de fluide respectif sont attribués à chacun des écoulements de fluide divisés (71, 72, 73, 74) et sont couplés à l'unité de commande (95).
3. Diviseur d'écoulement selon la revendication 1 ou 2, caractérisé en ce que l'actionneur (51; 91, 92, 93, 94) présente une résistance hydraulique modifiable de façon continue.
4. Diviseur d'écoulement selon l'une quelconque des revendications 1 à 3, caractérisé en ce qu'il permet la séparation en écoulements de fluide (31, 32; 71, 72, 73, 74) dans une gamme allant de 10 nL/min à 1 000 mL/min.
5. Procédé pour diviser un écoulement de fluide en un certain nombre d'écoulements de fluide divisés, en particulier pour la technologie de mesure d'un fluide analytique ou préparatif et/ou pour des systèmes microfluides, dans lequel une unité de commande qui est couplée à au moins un détecteur de travail, qui est attribué à un des écoulements de fluide divisés, et à un actionneur pour changer cet écoulement de fluide, régule la pression d'un des écoulements de fluide et/ou régule un des débits des écoulements de fluide, et dans lequel au moyen d'au moins deux détecteurs de travail (41, 42; 81, 82, 83, 84), le débit respectif des écoulements de fluide (31, 32; 71, 72, 73, 74) est mesuré, caractérisé en ce que lesdits détecteurs de travail se présentent sous forme de débitmètres massiques thermiques qui sont conçus de façon identique en ce qui concerne la dépendance de leurs signaux de mesure vis-à-vis des propriétés physiques du fluide circulant à travers le diviseur d'écoulement, dont respectivement au moins un est attribué à chacun des écoulements de fluide divisés (31, 32; 71, 72, 73, 74), où l'effet de dépendance des signaux de mesure des détecteurs de travail (41, 42; 81, 82, 83, 84) vis-à-vis des propriétés physiques du fluide est compensé au moyen d'un développement proportionnel des signaux de mesure des détecteurs de travail (41, 42; 81, 82, 83, 84), et dans lequel l'unité de commande régule les débits des écoulements de fluide (31, 32; 71, 72, 73, 74) de sorte que le rapport entre au moins deux des débits des écoulements de fluide (31, 32; 71, 72, 73, 74) reste constant.

EP 1 248 096 B1

Fig.1

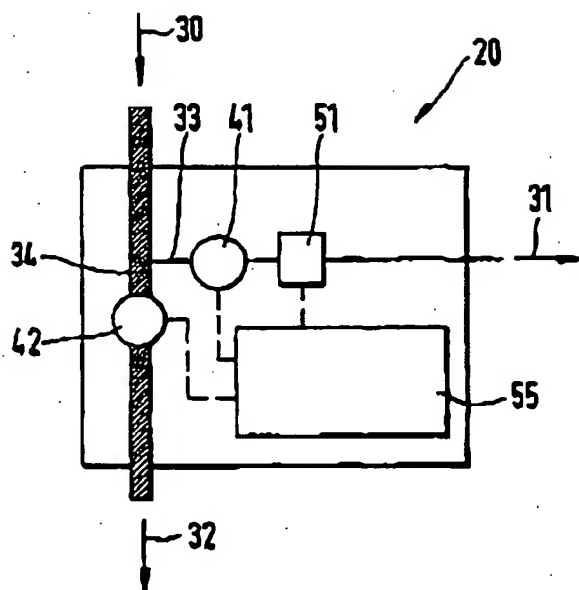
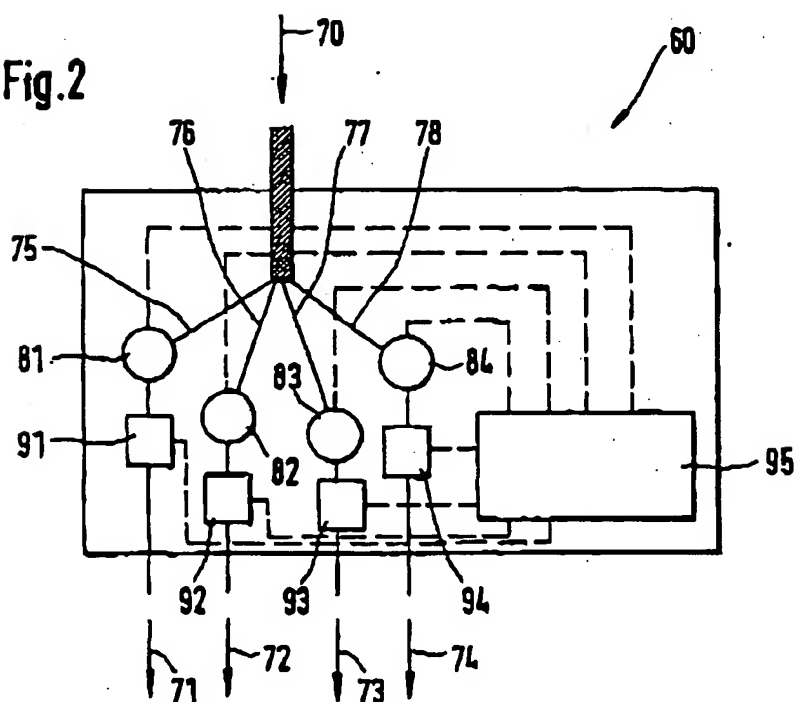


Fig.2



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